

## PATENT SPECIFICATION

DRAWINGS ATTACHED

L165.312



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## COMPLETE SPECIFICATION

## Helically Crimped and Crimpable Composite Staple Fiber

We, E. I. DU PONT DE NEMOURS AND COMPANY, a Corporation organised and existing under the laws of the State of Delaware, United States of America, located at Wilmington, State of Delaware, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to helically crimpable or crimped composite staple fibre and is an improvement in or modification of the invention claimed in Patent Specification No. 1,075,689.

Patent Specification No. 1,075,689 describes and claims a helically crimpable composite filament comprising a laterally eccentric assembly of at least two synthetic polymeric components at least the first of which is a polyester partly crystalline in stable conformation that does not exceed 90 per cent of the length of the fully extended molecular conformation of the said polyester, the remaining component or components having a lower shrinkage than the first polyester component, the said polyester being adapted to assume generally a position on the inside of crimp helices formed when the assembly crimps.

The present invention provides a helically crimpable or crimped composite staple fiber comprising a laterally eccentric assembly of at least two synthetic polymeric components at least one of which is a polyester partly crystalline in stable conformation that does not exceed 90 per cent of the length of the fully extended molecular conformation of said polyester, the said polyester generally assuming a position on the inside of crimp helices formed when the assembly crimps, the staple fiber having a crimp index less than 8 per cent, a crimp development of from 5 to 15 per cent and a crimp frequency of from 9 to 15 crimps per extended inch.

The present invention also embraces yarns and fabrics made from the staple fiber described immediately above. The term "stable" means that the polymer retains its crystalline conformation under all conditions which do not melt the polymer.

In a preferred embodiment the components are eccentrically coextensive throughout the length of all filaments, and are present in a substantially constant ratio in the filament cross section. In another embodiment each filament has a substantially constant ratio of the components arranged eccentrically throughout its length, but the ratio of the components present is varied from filament-to-filament. In still another embodiment, the components vary in their cross sectional ratio both filament-to-filament and along a given filament. Each of these embodiments has characteristics which are peculiar thereto, but all have in common the advantages of practical crimpability against restraining load imposed by woven fabrics made therefrom, good stretchability and moderate-to-high-power recovery from stretch.

Both components may comprise polyesters having a non-extended crystalline conformation or even "non-extended" polyesters having the same chemical composition if care is taken in selection of other polymer characteristics. A difference in molecular weight between components or use of a homopolymer for one component and a copolymer for the other, as examples, can provide the necessary differential in shrink-ability and, thereby, spontaneous crimpability. It is preferred, however, that the second component, that is, the component which is located on the outside of the helix when the fiber is crimped, comprises a polymer which is more extended in its crystalline conformation than is the polymer of the first component. Even more desirable products result, generally, if the second component crystallizes in a conformation in which the crystalline repeat-distance is 95% or more

that of the fully extended chemical repeat distance.

The crystalline conformation of the component which is inside of the helix of the crimped, composite filament, as has been mentioned above, must be non-extended to the extent that the crystalline repeat-distance is 90% or less that of the fully extended chemical repeat-distance. Although the reasons for this requirement are not fully established, and the invention should not be limited by a theoretical consideration, the discussion which follows may assist in understanding the scope of the invention.

Spontaneous crimping of a bicomponent fiber is the result of differential length changes between components which arise from a difference between components in retraction-from-draw, shrinkage, or both; these, in turn, are due to molecular disorientation involving bond rotation leading to molecular conformation changes. The forces leading to such changes arise from directionally unbalanced kinetic energy of bond rotation as a result of molecular orientation during drawing. There is essentially no phenomenological distinction between retraction-from-draw and shrinkage. The distinction is found only in the environment required for the effect to occur. Retraction-from-draw is fully analogous to retraction from stretch in an elastomer in that it occurs at room temperature. The crimp resulting from a differential in retraction-from-draw, therefore, will develop upon unwinding a yarn from its package and allowing it to relax. Shrinkage refers to that contraction in length which occurs when an oriented fiber is for the first time heated under conditions of low restraint (or none) to a given temperature above ambient temperature. In general, both shrinkage and retraction-from-draw are enhanced by higher orientation in the amorphous zones of the fiber. Stable crystallites in the fiber cannot contribute to shrinkage, since there is no opportunity for bond rotation in a zone where, by definition, it is prevented by intermolecular order. The potential of a fiber component to shrink is affected, however, by changes in the degree of crystallinity. The direction of this effect, that is, to increase or to decrease shrinkage, is determined by the characteristic crystalline conformation of the polymeric component. If the polymer crystallizes in an extended conformation, (as do most polyesters), that is, one in which the length of the crystalline repeat-unit closely approaches that of the fully extended chemical repeat-unit, a unit added will increase the length of the crystallite more than its removal reduces the length of the amorphous region; thus the partially crystalline "extended-type" component loses amorphous orientation (and shrinkage) as it continues to crystallize. If, on the other hand, the added chemical repeat-unit, being of the non-

extended type, increases the length of the crystallite less than its removal reduces the length of the amorphous region, an increase in amorphous orientation results. Thus, further crystallization of such non-extended polymer components results in higher tendency to shrink.

From the above discussion, it follows that the unusual properties of the fiber of this invention are fully realized only if the overall orientation of the high-shrinkage component in the uncrimped fiber is greater than its orientation in its crystalline conformation. Thus it is desirable that this, the inner component, be the more highly oriented member.

Orientation of synthetic fiber may be accomplished in either or both of two ways: (1) by withdrawing the solidifying filament from the spinneret at a rate higher than its extrusion velocity and (2) mechanical stretching of the solidified filament. In a bicomponent fiber, it is obvious that the mechanical stretching step imparts the same draw ratio to both components and is therefore not highly effectual in establishing an advantage in orientation for the high-shrinkage component. To ensure that the advantage in orientation be accomplished, it is desirable that the high-shrinkage component be more highly oriented during spinning. This is conveniently done by using a higher molecular weight (higher melt-viscosity) polymer for the high-shrinkage component. This same effect can be realized, however, by suitable use of melt-viscosity adjuvants in one component, and such process variations are within the scope of this invention.

The use of a polyester which crystallizes in a non-extended conformation as the high-shrinkage component of a bicomponent fiber provides substantial advantages over a combination of polymeric components which shrink unequally due to another difference such as in molecular weight, tensile recovery, glass transition temperature, etc. While the composite fibers known in the art possess differing degrees of potential shrinkability among components as a result of variations in response of the components to manufacturing conditions employed, in no instance has such difference been found completely retained or, more strikingly, enhanced by stress and/or high temperature, as is the case with the products of this invention.

Maintenance of the critical crimp-potential (differential shrinkage and retraction between components) requires a good degree of geometrical stability intermolecularly in the high-shrinkage component, except for those rearrangements necessary to fiber shrinkage. Otherwise, the potential energy required for crimping may be dissipated by molecular rearrangements to a more stable condition (of lower shrinkage). It has been found that some polymers which crystallize in a non-

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- extended conformation fail to meet the requirements of the high-shrinkage component of this invention apparently due to unstable intermolecular geometry. It will be obvious that any intermolecular order instability ("slippage") will result in reduced total orientation whether it occurs in the crystalline or amorphous regions of the structure.
- Accordingly and subject to the limitation that at least one component be a polyester partly crystalline in stable conformation not greater than 90% of the length of the fully extended molecular conformation, it is apparent that composite fibers of the present invention can be any of the materials heretofore employed in composite or other fibers. Condensation polymers and copolymers, e.g. polyesters, polyamides and polysulfonamides, and especially those that can be readily melt spun are preferred for both components. Suitable polymers can be found, for instance, among the fiber-forming polyesters and polyamides which are described in British Specification No. 461,236 and U.S. Patents Nos. 2,071,250, 2,465,319, 2,190,770, 2,130,523 and 2,130,948. The polyesters that are preferred for the component exhibiting the critical stable conformation are poly(trimethylene terephthalate), poly(tetramethylene terephthalate), poly(trimethylene dinaphthalate)\* and poly(trimethylene bibenzoate). Poly(ethylene terephthalate) is the preferred polyester for the other component, but other polyesters such as the corresponding copolymers of ethylene terephthalate containing sebacic acid, adipic acid or isophthalic acid as well as those containing recurring units derived from glycols with more than four carbon atoms in the chain can be used as well. Preferred polyamides comprise, for example, such polymers as poly(hexamethylene adipamide), poly(hexamethylene sebacamide), poly(epsilon caproamide) and copolymers thereof.
- \* Trimethylene dinaphthalate throughout the application means trimethylene 2,6-dinaphthalate.
- The conformations of a number of polymers in their crystallites have been deduced from X-ray and model data. Table I gives the chemical and crystalline repeat-distances for a number of polymers:

TABLE I  
Repeat-distances (Å)

Polymer repeat-unit	Chemical	Crystalline	% Extended
ethylene terephthalate	10.9	10.7	98.
trimethylene terephthalate	12.2	9.1	75
tetramethylene terephthalate	13.4	11.7	87
trans-cyclohexanedimethylene terephthalate	14.7	14.2	97
ethylene 2,6-dinaphthalate	13.4	13.1	98
trimethylene dinaphthalate	14.5	11.5	79
trimethylene bibenzoate	16.6	13.3	80
1,3-cyclobutane dimethylene terephthalate	14.3	13.4	94
1,3-cyclobutane dimethylene bibenzoate	18.6	18	97
hexamethylene adipamide	17.4	17.2	99

Determinations of this nature are accomplished as follows: Measurement of the "% Extended" parameter is done as follows (the order of steps A and B is immaterial):

5 *Step A—Measurement of Crystalline Repeat-Distance.*

A parallel bundle of oriented and partly crystalline fibers is mounted in an X-ray beam with the fiber axis perpendicular to the beam. A flat photographic film is placed in and perpendicular to the X-ray beam at a distance of  $a$  mm. from the fiber array on the opposite side from the X-ray source. The film is suitably exposed and developed to show a "fiber pattern" consisting of a family of more-or-less complete hyperbolae with its axis parallel to the fiber axis, i.e., on the so-called meridian. The distance on the film along this line from the primary-beam image to each hyperbola is measured and designated  $e_n$ ,  $n$  being the ordinal number of the layer line counting away from the equator as zero. The diffraction angle  $u_n$  is defined as

$$u_n = \tan^{-1} e_n/a.$$

25 The identity period (Crystalline Repeat) is then simply calculated from

$$\text{I.P.} = \frac{n\lambda}{\sin u_n}$$

where  $\lambda$  is the wavelength of the X-rays used. The above notation follows G. L. Clark, "Applied X-rays", McGraw-Hill, New York (1955), p.401. The patterns from various polymers and particular values of  $e_n$ , of course, differ.

35 *Step B—Measurement of Chemical Repeat-Distance.*

A molecular model of the polymer in question is made from a commercially available scale-model kit such as the "Dreiding" kit manufactured by W. Buchi, Flawil, Switzerland. The interatomic bonds along the chain are rotated so as to give the longest straight length along the molecular chain. The distance from any nucleus in the chain to the corresponding nucleus in the next repeat-unit is measured, converted to Angstrom units, and designated the Chemical Repeat of the polymer.

*Step C—Calculation of % Extended.*

50 The Crystalline Repeat-Distance from Step A (which is in Angstrom units) is divided by the length calculated from measurements in Step B. The result is multiplied by 100 to give the per cent extended.

55 If the result of Step C is greater than 100%, obviously the crystal repeat-unit consists of more than one chemical repeat-unit.

The actual number can sometimes be estimated from geometrical considerations or from a more detailed analysis of the X-ray pattern. Since the crystal repeat must be an integral number of chemical repeats, assigning one chemical repeat therefore gives the maximum possible extension: if there were two chemical repeats, the per cent extension would be halved, etc.

The art of composite filaments, to which the present invention generally relates, is well developed and reference may be made to techniques already known for application to the present discovery. A partial list of U.S. patents that may be referred to for this purpose includes Breen, 2,987,797; Radow et al, 3,039,174; Breen, 3,038,236; Taylor, 3,038,237; and Zimmerman, 3,038,235; as well as British Specification No. 805,033. In addition, the various spinnerets described in those references as well as the manner of using them may be used in this invention. Others can, of course, be employed and one suitable type is indicated in the attached drawing in which:

Figure 1 is a schematic, transparent view of a spinneret plate of the post-coalescence type;

Figure 2 is a side view of cooperating channels in a spinneret assembly;

Figure 3 is an enlarged view showing the face plate openings of channels of the type of Figure 2; and

Figures 4, 5 and 6 are cross-sections of some of the type filaments that can be produced in this invention.

Filaments are produced with the indicated type spinneret by conventional procedures. Accordingly, and referring to Figures 1, 2 and 3, by means not shown, two melts are separately metered into the two rings of holes designated as 1 and 2, in Fig. 1 in spinneret 4. A sealing means (not shown) prevents mingling of the two melts at the back face 3 of spinneret 4. The two melts flow through individual channels 5 and 6 to the front face 7 of spinneret 4 where they merge into a side-by-side composite filament as they leave the spinneret assembly.

The filament generally is withdrawn from the spinneret at a speed that attenuates the filament, and is thereafter drawn. The conditions applied for drawing the spun multi-component filaments of this invention may vary in wide limits. In addition to the processes of drawing described in some of the examples given hereinafter, a hot pin may be used, or the yarn may be passed over heated rolls, as additional examples. In general, the precise amount of draw is established by use of feed and drawing rolls which are driven at the appropriate differential in speed, care being taken to assure that the yarn doesn't slip on either. Two methods for assuring positive control of speed

which are appropriate for feeding to or with-drawing from a drawing zone are described in U.S. 3,101,990.

Also, the temperatures at which the fila-  
 5 ments are drawn may vary in wide limits and depend mostly upon the properties of the single materials forming the composite fila-  
 10 ment and of the final desired results. As is the case in the production of conventional unitary filaments, the preferred drawing tem-  
 15 peratures for the composite filaments of this invention may vary between room tempera-  
 20 ture and slightly elevated temperatures; for example temperatures of about 100°C or  
 25 somewhat higher may be used. Since in the present invention combinations of at least two different materials usually are employed, the specific drawing characteristic of each material should be considered in order to obtain  
 30 best results. Drawing temperatures which are lower than the glass transition temperature ( $T_g$ ) of the several components may be em-  
 35 ployed where a separate plasticizing step is provided. Moreover, if desired the drawing and taut heat treatment may be coupled in  
 40 a continuous process to obtain the desired orientation and crystallization.

The filaments can thereafter be treated by  
 30 crystallizing under conditions wherein no shrinkage can occur. In other words the crystallization is effected under conditions of tension which equals the forces developed in the filaments during the treatment. Crystallization or length stabilization respectively of  
 35 many of the fiber-forming high polymeric materials can therefore be accomplished preferably by a heat treatment of the taut filaments. The temperature applied should generally be higher than the apparent minimum  
 40 crystallization temperature of the higher shrinkage component which is well known or can easily be determined for each polymer. A convenient method for determining the apparent minimum crystallization tem-  
 45 perature ( $T_i$ ) is described, e.g., in British Specification No. 695,567. Preferably, however, the apparent minimum crystallization temperature is determined by X-ray diffraction measurements on samples of filaments which  
 50 have been drawn in cool water to prevent crystallization and which have been subjected to taut heat treatment at progressively increasing temperatures. Filament exposures are suitably made on a Hilger semi-micro-focus  
 55 diffraction unit using a flat plate Norelco micro-camera similar in design to that described by Fankuchen and Mark, J. Applied Physics 15,364 (1944). The degree of crystallinity may be judged by direct examination  
 60 of the diffraction pattern or from radial densitometer traces along the equator of the X-ray diagram. Such a trace will show distinct peaks for fibers having a well developed crystalline structure whereas with an amor-  
 65 phous structure or with very low degrees of

crystallinity the peaks cannot be resolved. The apparent minimum crystallization temperature by this method is the minimum temperature of heat treatment at which a definite crystalline structure is detectable from direct examination of the X-ray diagram or at which distinct peaks are observable in the densitometer trace.

It is the important characteristic of those  
 75 polyesters having the crystalline repeat distance 90% or less than the chemical repeat distance that largely contributes the enhanced and highly unusual properties of composite  
 80 fibers in the present invention. For the composite fibers known heretofore, heat treatment at constant length tended to destroy, or at least lessen, shrinkage characteristics. That happens in the present invention also, but  
 85 only with respect to the components crystallizing in the fully (or more fully) extended form. For the indicated special polyesters, such heat treatment enhances the shrinking characteristic. Consequently, with a preferred  
 90 combination in the composite assembly, the shrinkage differential of the two components will ordinarily be greater after the taut heat treatment than before.

Therefore, any treatment which meets the  
 95 requirement of effecting crystallization of the low shrinkage component may be used in annealing fibers in this invention. A taut heat treatment at a temperature high enough and for a time long enough to provide crystallization of the low shrinkage polymeric component is preferred. The temperatures applied  
 100 in this heat treatment will generally be higher than the apparent minimum crystallization temperature ( $T_i$ ) of the component which is to be crystallized, which is defined as the lowest temperature at which the fiber may  
 105 be treated to produce a substantial degree of crystallinity in its structure. Only a short period of time at the annealing temperature is needed, for example only a fraction of a second. Extended annealing times are not deleterious, but neither are they normally  
 110 economic, so they are not used. In the examples given hereinafter, the term "annealing" indicates that the yarn was exposed to the indicated temperature for about 0.1  
 115 seconds or longer while held at constant length.

The filaments as produced upon taut an-  
 120 nealing may be used as such and crimp may be developed in the ultimate product. Alternatively, crimp may be developed first, and the crimped product then used. Any conventional hot relaxing step now known in the art may be employed to develop the char-  
 125 acteristic helical crimp.

In addition to the side-by-side species produced in the exemplified spinneret, it might  
 130 sometimes be desirable to spin a bundle of filaments which comprises composite filaments containing the components in various

ratios through one and the same spinneret. An example is a bundle of two-component composite filaments which comprises filaments consisting of 20% by weight of the higher shrinkage component and 80% by weight of the other, a 30%/70% ratio, a 40%/60% ratio and a 50%/50% ratio, respectively. Such filament bundles containing composite filaments with various ratios of components can very conveniently be produced by utilizing the spinneret which is shown in Figures 6 and 7 of the U.S. patent to Breen 3,118,011. As a generality and without regard to the type composite fiber being produced, the non-extended component usually comprises 20 to 80% of the composite and the other components comprise the remainder. The denier of the resulting product will be that usually produced in this general art, and is not of significance to the invention.

Low crimp fibers made according to this invention are particularly useful in the form of staple fibers in which they possess many of the attributes of wool and can be used to produce bulky fabrics with wool-like tactile aesthetics either alone or in fiber blends with wool and other man-made fibers.

The inherent differences in characteristics of and textile processes leading to staple-spun yarns, as compared to continuous filament yarns, are reflected in differing requirements for the fibers which constitute those yarns. High-crimp filament yarns make attractive, bulky and aesthetically pleasing fabrics. Staple fibers with similar crimp properties are boardy, harsh and aesthetically undesirable in typical worsted yarn and fabric constructions. As will be illustrated, lower crimp frequency (CF) and crimp development in staple fibers lead to attractive, bulky, wool-like fabrics. CF is measured as crimps per extended unit length after boil-off under 1.5 mg./den. load. These crimp properties may be lowered in a variety of ways, including reduction of molecular weight (hence orientation) of the inner, non-extended, component relative to the outer component; reduction of the ratio of the inner to the outer component in the fiber cross section; changes in the disposition or configuration of the two components in the fiber; reduction of processing temperatures, draw-ratio, or time of exposure to elevated temperatures during constant-length processing of the filaments; controlled or free relaxation of the fiber at elevated temperatures during processing; and by other methods which will be apparent to those skilled in the art.

Another required property of staple fibers which is relatively unimportant in filament yarns is the initial crimp as estimated by crimp index (CI). In this specification, CI is measured in a manner identical to that for CD except that the boil-off step is eliminated. Thus, CI is a measure of the crimp available

during processing of the fiber to yarn. The amount of initial crimp has a pronounced influence on the efficiency of processing a staple fiber to a spun yarn. Excessive crimp in fiber intended for cotton-system or worsted-system processing leads to non-uniform carding and neppy, poor quality yarns. In accordance with this invention the crimpable staple or tow has  $CI < 8$ ,  $9 < CF < 15$ , and  $5 < CD < 15$ . Such fibers may be processed either as staple or as tow, the resulting yarns offering knitted and woven fabrics of good uniformity, high bulk and wool-like tactility.

While CI is an important parameter for controlling processibility and CF plays a major role in determining ultimate aesthetics, CD is a major determinant of fabric bulk. Maximum bulk is attained in fabrics when most of the crimp is developed in yarn or fabric, as opposed to staple or tow, form. Development of bulk in a yarn or fabric depends upon the crimp-ability of the fiber at elevated temperatures under the restraints imposed by the yarn or fabric construction. It is characteristic of the fibers of this invention that while tensions up to 1 gpd. may reduce the initial crimp (CI) of the fiber, such tensions have little or no effect on the ultimate, or developable, crimp (CD). Thus, it is possible to start with a tow of, say, 10% CI, 8% CD, and to process that tow through a Pacific converter, pin drafting, roving and spinning to produce a yarn in which the fibers have CI of about 5% but a retained CD of 8%. Such fibers would be considered "crimpable" in this context even though originally CI exceeded CD. A convenient test of crimpability in this context may be carried out by measuring CI and CD as previously described but subjecting the fibers to a load of 1 gpd. for 30 minutes before measuring CI and then boiling off the fiber and measuring CD in the normal manner. The staple or tow fiber will be classified as crimpable if CD is greater than CI after this procedure.

It is also important that crimp frequency be stable to (i.e., resilient under but not appreciably changed by) the loads, temperatures and plasticizing agents the fibers may be subjected to in processing and ultimate use. In a crimp-frequency-stable fiber CF as measured in the test corresponds to that of the surface fibers (which largely determine tactility) after, for example, yarn or fabric boil-off (where loads encountered by the external fibers would be significantly lower than those encountered by internal fibers or in the test), heat setting, or dyeing. Such stability appears as a unique advantage of fibers of this invention, which rely on differences in crystalline conformation. Typical bicomponent fibers of the art, crimp to varying frequency depending on environmental conditions to which they are subjected.

While most of the immediately foregoing discussion and the following examples are related to woolen and worsted staple processing and fabrics, similar advantages can be attained in blends with cellulose and other fibers blended with suitably crimped and/or crimpable staples of this invention and processed, e.g., on the cotton system.

In the following examples, except as otherwise indicated, the terms employed in evaluating polymers and fibers have the following meanings:

*Relative Viscosity* refers to the ratio of the viscosity of a 10% solution of the polymer in a mixture of 10 parts of phenol and 7 parts of 2,4,6-trichlorophenol (by weight) to the viscosity of the solvent itself, both measured at 25°C. and expressed in the same units.

*Intrinsic Viscosity* is defined as the limit of the fraction

$$\frac{\ln(r)}{c}$$

as concentration  $c$  approaches zero, where  $r$  is the relative viscosity as defined above, except that relative viscosity is measured at several concentrations to facilitate extrapolation to zero concentration, and the solvent employed in this measurement is a mixture of three parts of methylene chloride and one part of trifluoroacetic acid (by weight). A more detailed discussion of methods of measuring relative and intrinsic viscosities is given in *Preparative Methods of Polymer Chemistry*, Sorenson & Campbell, Interscience, 1961.

*Skein Shrinkage* is determined by the following procedure:

1. From the known denier of the yarn, calculate the number of turns of a skein reel required to achieve a denier of 1500 (167 Tex) (the circumference of the reel may be any convenient length), using the formula

$$T = \frac{1500}{d}$$

where  $T$  designates turns on the skein reel and  $d$  is denier of the yarn; round off to the nearest even number of turns. Prepare and label a skein from each yarn to be tested. It will be obvious that such a skein must be considered as 3000 denier (334 Tex) when loaded as a loop.

2. Hank the skein and apply a 300-gm. weight at the bottom of the loop. Exercise gently four times. Wait 15 seconds and measure initial length of the skein ( $L_o$ ).

3. Replace the 300-gm. weight with a 4.5-gm. weight and immerse the skein in boiling water for 15 minutes. Remove from bath.

4. Dry the skein overnight hanging under

its own load. Exercise and measure crimped length ( $L_c$ ) as in 2 above.

5. Reapply the 300-gm. load, exercise and measure extended skein length as in 2, above ( $L_e$ ).

6. Calculate skein shrinkage by the formula

$$SS = \frac{(L_o - L_c)100}{L_o}$$

*Crimp Development*: Calculate from data obtained in the Skein Shrinkage procedure by the formula

$$CD = \frac{(L_o - L_c)100}{L_e}$$

*Crimp Elongation*: Determine by the procedure employed in Skein Shrinkage with the addition of one more step: after measurement of  $L_o$  (Step 5), the 300-gm. load is replaced by the 4.5-gm. load and a second measurement of recovered length under the smaller load is made as in Step 2 of that procedure ( $L_r$ ). Crimp Elongation is calculated by the formula

$$CE = \frac{(L_o - L_r)100}{L_r}$$

#### EXAMPLE 1

This example illustrates the utility of the fibres of this invention in preparation of worsted fabrics of improved bulk and tactility.

A 98/2 mol-ratio copolymer of PPT and ethylene sodium sulfo-isophthalate of 0.63 intrinsic viscosity and a 98/2 mol-ratio copolymer of PET and ethylene sodium sulfo-isophthalate of 15.0 relative viscosity are spun in a 35/65 ratio to side-by-side round bicomponent fibers as in Example V of the main case, Patent No. 1,075,689 (Patent Application No. 31018/65), except that extrusion temperature is 290–295°C. and wind-up speed is 530 ypm (485 m./min.). The filaments are drawn to 316% of their spun length in a bath of water at 83°C. and annealed at constant length at 180°C. for 30 seconds. Tow properties are: tenacity 2.2 gpd, elongation 21.1%, CI 3.6%, CD 13.5%, CF 9.6 crimps/extended inch (3.8 crimps extended cm.) and SS 2.5%.

A 1/27 worsted count yarn is spun on the worsted system to comprise 55% of the fiber of this example (which has been cut to 3" (7.6 cm.) staple) and 45% 64–70's wool. The yarn is woven to a 2 × 2 twill and mill-finished in parallel with a similar fabric comprising 55/45 commercial polyester staple/wool. An improvement in bulk (ASTM bulk 2.40 vs. 2.08 cc./gm.) is observed for the bicomponent-fiber blend fabric, which in addition has a pleasant, more wool-like handle.

## EXAMPLE 2

- This example illustrates the effect of varying annealing conditions on crimp properties in the range preferred for staple. A 50/50-ratio bicomponent staple is prepared with a PET polymer of 20 relative viscosity as sheath and a PPT of 0.8 intrinsic viscosity as an eccentric core, using a spinneret such as described as Figure 1 of the Breen U.S. Patent 2,987,797, a spinning temperature of 267°C., and a wind-up speed of 1000 ypm. (915 m./min.) to yield a yarn denier of 1150, 120 filaments. Bobbins of the spun yarn are combined to form a 37,950-denier tow, which is drawn in water at 90°C. to 330% of its spun length and annealed on rolls at a series of temperatures, while stretching an additional 10% during a 30-second exposure. The results are summarized in Table 12.

TABLE 12

Crimp properties, as measured at 1.5 mg/den load

Item	Anneal. Temp. °C.	SS%	CI, %	CD, %	CF, cpi
A	160	6	8	19	16
B	140	8	9	13	18
C	120	9	10	10	16
D	None	15	2	3	6

- Bulky fabrics may be obtained from Item D by two different techniques as illustrated by the following test. Item D is processed on the worsted system (as 3.5-inch (8.9 cm.) staple) to 20/1 cotton-count 55/45 polyester/wool blend yarn with 18 tpi, then woven to a 2 × 2 twill fabric having a weight of 6.5 oz./sq. yd (222 gms./sq. meter) after finishing. The fabric is divided and finished by two methods. Part 1 is scoured at the boil, dyed, lightly fulled and heat-set at 160°C. for 3 minutes. Part 2 is similarly finished except that the fabric is heat-set at 160°C. for 5 minutes, while being held at constant length and width, prior to the initial boil-off. This fabric annealing step approximates that of the annealing given Item A yarn.
- Both parts of Item D fabric are bulky, but the bulk of Part 1 fabric is characteristic of that obtained by a mixed-shrinkage mechanism; it has a soft hand and a somewhat fuzzy surface. Part 2 fabric, on the other hand, comprises filaments of spiral crimp, and has a more resilient hand, more even surface and a desirable degree of stretchiness, which characteristics are attributable to the helical crimp of the bicomponent staple. These characteristics are enhanced rather than lost, as a result of taut annealing before boil-off.

## EXAMPLE 3

- A series of PPT/PET staples is prepared by means taught in the preceding examples to have a range of crimp frequencies. They are blended in 70/30 ratio with wool, processed on the worsted system to 1/30 worsted count yarns with 14 tpi and knitted on a 20-cut machine to tubing which is finished with a boiling scour, piece dyed and tumble-dried. Using the method as described below, the fabrics are analyzed for fabric-on-fabric kinetic friction ( $F - F_k$ ). Results are summarized in Table 13, with, as comparison items, similar fabrics comprising wool and a bicomponent fiber more typical of those of the prior art.



TABLE 13

Fiber CF	F—F <sub>k</sub>	Tactility**
5	0.76	slick-lean
9	0.87	wool-like
11	0.87	"
12	0.93	"
14	0.93	"
17	1.01	harsh, wool-like
21	0.99	" "
9 (wool)	0.91	—
52*	1.67	harsh

\*Fabric prepared in a similar manner from side-by-side bi-component fiber comprising PET and a 85/15 copolymer of PET and poly(ethylene isophthalate) in a 50/50 cross-sectional ratio.

\*\*Subjective evaluation.

5 *Fabric-to-fabric kinetic friction (F—F<sub>k</sub>) is determined by measuring the force required to move one flat fabric sample of 2.63 square inches area (17 cm.<sup>2</sup>) at a speed of 3.5 cm./min. relative to another sample of the same fabric, the two being forced into intimate contact with each other by a load of 110 gms. The procedure is as follows:*

10 The fabric samples used in this test are typically single-thickness knitted fabrics, although woven fabrics may be used with proper calibration. One sample of fabric is laid over a large flat surface which can be moved horizontally by a lead screw. The top of this surface is covered by sandpaper, sand-side up, firmly attached thereto. The sandpaper serves to prevent slipping of the fabric. 15 A wooden block, similarly bearing on one of its faces a piece of sandpaper carries the second sample of fabric, which is carefully wrapped around and attached to the block to provide a definite flat area of exposure. The block carries a hook on the center of one side face which can be attached to a strain gage. 25

The block, bearing the second fabric sample is laid fabric-to-fabric over the first sample and the strain gage attached to the hook. 30 Weights are added to the block to result in a fabric-to-fabric loading of 110 gms., the total area amounting to 2.63 in.<sup>2</sup> (17 cm.<sup>2</sup>).

The lead screw is turned at a steady rate to result in horizontal movement of the first

sample of fabric at the rate of 3.5 cm./min. 35 The force developed to hold the loaded block stationary over the moving fabric sample is read and results calculated as follows:

$$F - F_k = \frac{\text{developed force (gms.)}}{\text{load on sample (gms.)}}$$

#### EXAMPLE 4

40 This example illustrates utility of a sheath-core bicomponent fiber comprising PET and poly(tetramethyl terephthalate) (PBT) in staple form.

45 A 50/50 PET/PBT bicomponent yarn is prepared with a 21 relative viscosity PET as sheath and a 0.95 intrinsic viscosity PBT as eccentric core using a process similar to that of Example 2. Spinning temperature is 285— 290°C., windup speed 500 ypm (457 m./min.), yarn count 1200 denier, 120 filaments. 50 Seventy-five bobbins are combined as a tow, which is drawn to 371% of its as-spun length in water at 90°C. It is annealed on rolls at 180°C. for 33 seconds while stretching an additional 10% of its drawn length. The resulting fiber exhibits SS 3%, CI 14%, CD 17%, and CF 14 cpi. 55

The tow is cut to 3.5 inch (8.9 cm.) staple and processed to 55/45 polyester/wool yarns with 20/1 cotton count and 14 tpi. Processibility is found to be satisfactory despite the rather high crimp. Crimp development and 60

bulk of the boiled-off wool blend yarns are similar to those of Item A, Example XXI, or of Example XX.

#### EXAMPLE 5

- 5 A series of 3 inch (7.6 cm.) staples is prepared in a manner paralleling that of Example 1, except that annealing is varied to

produce a range of CI. Table 14 summarizes the results of carding these staples. As will be seen, a CI of about 8 is borderline-to-satisfactory for this method of textile processing. CI of 5 or less is preferred. It has been found, however, that higher CI tow (about 9%) processes in a satisfactory manner on the Pacific Converter to yield low-nep-level yarns.

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TABLE 14

Fiber CI—%	Card Web Appearance
3.0	no neps
3.9	no neps
5.5	no neps
7.5	excessive neps
7.9	no neps
8.3	excessive neps

#### EXAMPLE 6

- 20 A side-by-side bicomponent fiber comprising 65% of a 98/2 mol-ratio copolymer of PET and ethylene sodium sulfoisophthalate of 12.5 relative viscosity containing 0.25% tetraethyl silicate and 35% of a 98/2 mol-ratio copolymer of PPT and ethylene sodium sulfoisophthalate of 0.63 intrinsic viscosity is spun
- 25 at 285°C. from a 120-hole spinneret. The filaments are quenched with room-temperature air and wound-up at 525 ypm (480 m./min.) to give a spun denier of 12 dpf. Yarn from 56 packages of 1440 denier each, is
- 30 drawn to 407% of its as-spun length in water of 85°C. to yield a nominal 3 dpf tow, which is annealed at constant length by exposure to rolls heated to 180°C. for 15 seconds. It is mechanically crimped to 8—10 cpi (3.1—3.9 cpm) and cut to 3-inch (7.6 cm.) staple. Properties are: tenacity 2.5 gpd., elongation 18%, CF 9 cpi, CD 10%, CI 3.7% and fiber shrinkage 2.9%.

- 40 The staple is blended 50/50 with a commercially available, basic-dyeable polyester staple intended for use in pill-resistant fabrics and processed to 2/30 worsted count yarn with 12 tpi Z-twist in the singles and 6 tpi S-twist in the plied yarn. The yarn is knitted
- 45 at 13 courses/inch (5.1 courses/cm.) on a 12-cut circular knitting machine. The resulting fabric is piece-dyed at the boil using a disperse dye formulation comprising 5 g./liter of a carrier consisting of equal parts of dimethyl terephthalate and benzanilide. Bulk is

developed during dyeing. The finished fabric has excellent stitch clarity and good bulk, resilience and liveliness. Tactile aesthetics very closely approach those of an all-wool counterpart. The test fabric has a weight of 8.63 oz./yd.<sup>2</sup> (292 gms./m.<sup>2</sup>) and bulk of 5.47 cc./g. under a standard 3 g./cm.<sup>2</sup> load and 4.37 cc./gm. under a load of 40 g./cm.<sup>2</sup>.

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#### EXAMPLE 7

This example illustrates the advantage of delaying the development of the major portion of the bicomponent crimp until the fiber is in yarn or fabric form.

Bicomponent staple is prepared substantially according to the procedure of Example 6 to have CI 3.7% and CD 25—30%, (as measured under no boil-off load). Card sliver is prepared; it is used to prepare worsted yarns by each of three process sequences:

A. The sliver is blended 70/30 with wool in pin-drafting and spun to 1/30 worsted count yarn with 14 turns twist.

B. The sliver is relaxed at 100°C. to develop the crimp, blended 70/30 with wool in pindrafting and spun to 1/30 worsted count yarn with 14 turns twist.

C. Roving prepared as in sequence A is steamed to develop crimp and spun to 1/30 worsted count yarn with 14 turns twist.

Crimp properties measured on single filaments of the various structures developed in this example are summarized in Table 16.

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TABLE 16

Structure	CI, %	CD, %*	CF, cpi*	Comments
Card Sliver A	5.4	28	10.5	
Roving A	6.5	27	11.1	
Worsted Yarn A	4.5	25	11.6	Good bulk
Card Sliver B	28	28	11.5	
Roving B	9.5	32	14.3	
Worsted Yarn B	11.2	23	12.6	Good bulk
Roving C	21	25	15	
Worsted Yarn C	17.5	14	11.9	Low bulk

\*CD and CF are measured after boiling off or steaming of the fibers in the form indicated. No restraining load is used during this relaxation.

Upon relaxing, yarn A bulks to about twice the diameter of yarn C. The basis for this is seen in comparison of CI and CD for these 5 yarns. Essentially all the developable crimp is already present in yarn C (17.5 CI vs. 14 CD), while the bulk of the crimp is yet to be developed in yarn A.

The bulk of yarn B is equivalent to that of yarn A after boil-off. This is explained by the fact that the crimp previously developed in sliver B is eliminated in pin drafting and is redevelopable in the yarn.

## EXAMPLE 8

15 This example illustrates properties obtained in bicomponent-fiber structures more repre-

sentative of the art.

Side-by-side bicomponent staple comprising as one component PET of 20 relative viscosity and as the second a 92.5/7.5 copolymer of bis(hydroxyethyl) terephthalate and bis(hydroxyethyl) isophthalate of 40 relative viscosity. The polymers are spun at a block temperature of 296°C. and wound up at 800 ypm (730 m./min.). The spun denier is 810, 60 filaments. A tow of 81,000 denier comprising such filaments is drawn to 400% of its spun length in a water bath at 90°C. and annealed on rolls for 33 seconds at a series of temperatures. Results are given in Table 30 17.

TABLE 17

Item	Anneal Roll Temp., °C.	Crimp Properties			
		SS	CI	CD	CF
A	180	4.5	1.5	11.5	12
B	160	6	1.7	12.6	14
C	140	10	1.4	13.3	18
D	120	20	4.5	13.8	28
E	Off	24	6.2	16.9	36

35 Note that as annealing temperature is increased, CD decreases, in contrast to the results obtained with the bicomponent fibers of this invention.

Items A, C and E are processed to 55/45 wool-blend worsted yarns of 20/1 cotton count

which are woven to 2 × 2 right-hand twill fabrics. The fabrics are finished in a standard manner for mill-finished worsteds. All fabrics are judged objectionably harsh, the harshness increasing with increasing crimp properties (A through E), although similar 40

5 fabric made from fibers of this invention with properties similar to item A had a pleasing tactility (e.g. Example 1). Microscopic examination shows the surface of the fabrics to be covered by tightly coiled fibers. It is postulated that the fibers coil on high temperature treatment during fabric heat-setting. This is confirmed by relaxing a sample of item E tow at tenter-frame temperatures 10 (150°C. or higher under essentially no restraint), whereupon the fiber develops more than 50 crimps per inch. Fibers of this invention maintain nearly their measured crimp frequency when subjected to fabric heat-setting conditions, as illustrated in Example XXVIII.

## EXAMPLE 9

This example further illustrates the unique stability of the helical crimp in bicomponent fibers of this invention when subjected to the plasticizing action of dye carriers. The importance of crimp frequency to aesthetics makes this characteristic a valuable contributor to the development of superior textile fibers.

Bicomponent staple is prepared in accordance with the procedure of Example 1. Crimp frequency before and after various treatments, as noted, are summarized in Table 18, as compared with fiber such as that illustrated by Example XXVII.

TABLE 18

Fiber	As Example 8		
	As Example 1	Sample A	Sample B
Crimp Frequency (cpi)			
Initial	12.5	13.8	13.8
After relaxed Boil-off			
a)	13.6	18	—
b) in 5 g/l Carrier A*	11.5	23	—
c) in 5 g/l Carrier B**	10.8	45	—
After relaxed heat setting at 155°C. for 40 seconds	13—15	—	32.7

\*Equal weights mixture of dimethyl terephthalate and benzanilide

\*\*isopropylidene 4,4'-diphenol

## EXAMPLE 10

35 This example describes the selective response of bicomponent fiber of this invention to fabric fulling, illustrating further the broad versatility of these fibers.

40 A PPT/PET staple is prepared substantially by the process of Example 1. The fiber is 3 dpf., 3½-inch (about 9-cm.) staple. Properties are: CI 4.2%, CD 21%, SS 3.1%, CF 12%.

45 The staple (55 parts) is blended with 45 parts 64—70's wool top through worsted carding, pin drafting, slubbing, roving and spinning to prepare 50's worsted count yarn of 17 tpi Z twist.

50 The yarn is doubled at 15 tpi S twist and woven to a 70 × 64 (warp/fill) 2 × 2 right hand twill fabric. The fabric is divided into two pieces, which are finished by desizing, scouring, drying, fulling, washing, dyeing and drying. The two pieces are handled in parallel

55 in all steps excepting fulling. Piece A is fullled at 206°F., while Piece B is fullled at normal room temperature.

60 Piece A is found to have a distinctly softer tactility. Microscopic examination reveals extensive fiber splitting to individual fiber components on the surface of Piece A, while Piece B exhibits no splitting.

## WHAT WE CLAIM IS:—

65 1. A helically crimpable composite staple fiber comprising a laterally eccentric assembly of at least two synthetic polymeric components at least one of which is a polyester partly crystalline in stable conformation that does not exceed 90 per cent of the length of the fully extended molecular conformation of said polyester, the said polyester generally assuming a position on the inside of crimp helices formed when the assembly crimps, the staple fiber having a crimp index less than 8 per cent, a crimp development of from 5 to 15

per cent and a crimp frequency of from 9 to 15 crimps per extended inch.

2. A staple fiber according to Claim 1 wherein a second component of said assembly has a crystalline conformation more closely approaching its fully extended molecular conformation than said first component.

3. A staple fiber in accordance with Claim 1 or Claim 2 in which the assembly contains as a second component poly(ethylene terephthalate).

4. A staple fiber in accordance with any one of Claims 1 to 3 in which said polyester is poly(trimethylene terephthalate), poly(trimethylene dinaphthalate), or poly(trimethylene bibenzoate).

5. A helically crimped composite staple fiber produced from the crimpable staple fiber of any one of Claims 1 to 4.

6. Helically crimpable, and helically crimped, composite filaments substantially as hereinbefore described in the Examples.

7. Yarns and fabric when prepared from the staple fiber of any one of Claims 1 to 6.

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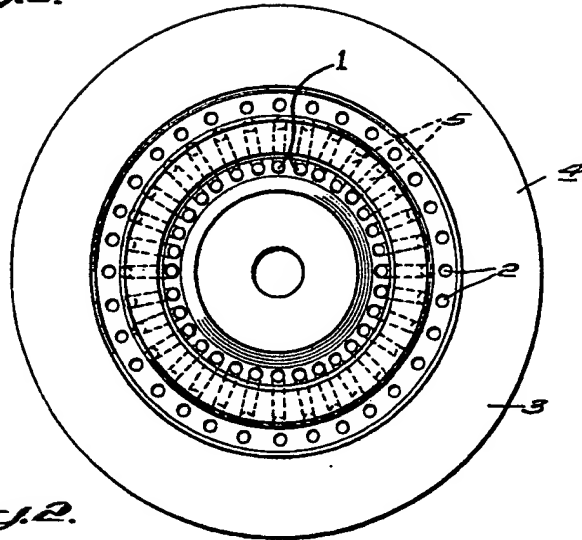
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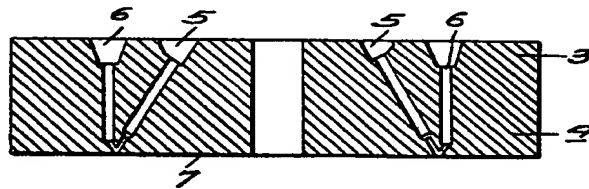
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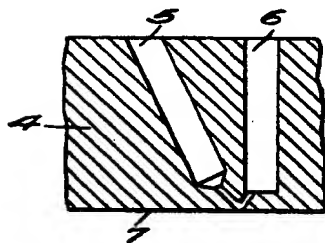
*Fig. 1.*



*Fig. 2.*



*Fig. 3.*



*Fig. 4.*



*Fig. 5.*



*Fig. 6.*

